TECHNICAL SERVICES DEPT

PROCESS ENGINEERING REVIEW

PRODUCT: XXXXXX
STAGE: XXXXX
LAB:

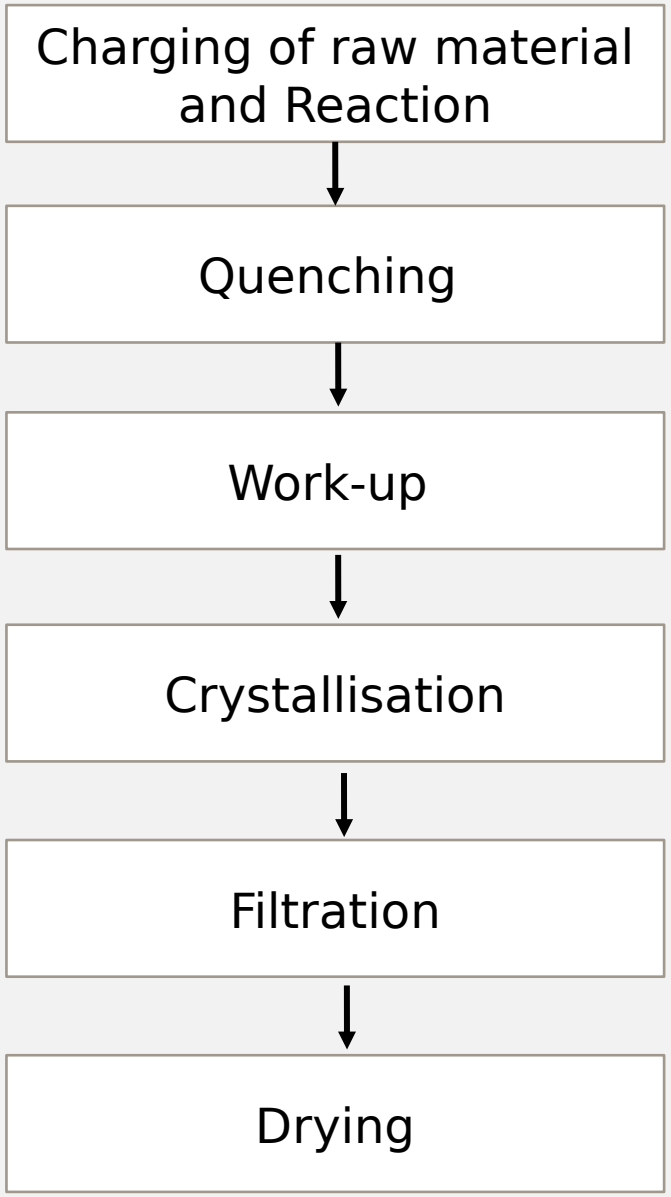
SCHEME AND STOICHIOMETRIC BALANCE

Reaction	$C_{13}H_{10}ClNO$	C_2H_7NO
Weight	1.00	3.03
Mol. Weight	231.68	61.08
No. Of Moles	0.0043	0.05
Chemical Name	2-AMINO-5-CHLOROBENZOPHE NONE	MONOETHANOL AMINE
Total Weight	4.03	
Mol. weight Balance	292.76	

→

$C_{15}H_{15}ClN_2O$	H_2O	Unreacted $C_{13}H_{10}ClNO$	Unreacted C_2H_7NO
1.08	0.07	0.1	2.8
274.75	18.01	231.68	61.08
0.00393	0.0039	0.00039	0.0457
ACPM ETHANOL	WATER	2-AMINO-5-CHLOROBENZOPHENONE	MONOETHANOL AMINE
4.03			
292.76			

PROCESS FLOW DIAGRAM



CHEMICAL INTERACTION MATRIX

	ACETONITRILE				
ACETIC ACID, GLACIAL	**Caution** ☐ Generates heat	ACETIC ACID, GLACIAL			
SODIUM BICARBONATE	**Compatible** ☐	**Caution** ☐ Generates gas Generates heat	SODIUM BICARBONATE		
METHYL TERT-BUTYL ETHER	**Compatible** ☐	**Compatible** ☐	**Compatible** ☐	METHYL TERT-BUTYL ETHER	
ETHANOL	**Compatible** ☐	**Caution** ☐ Flammable Generates gas Generates heat Intense or explosive reaction	**Compatible** ☐	**Compatible** ☐	ETHANOL
WATER	**Caution** ☐ Flammable Generates gas Toxic	**Compatible** ☐	**Caution** ☐ Generates gas	**Compatible** ☐	**Compatible** ☐

Note: *Above data is obtained from* https://cameochemicals.noaa.gov/search/simple

PROCESS ENGINEERING STUDIES

1) Reaction:
   a) Basic observations (Nature of mass, volumes, solvent / reagent used, etc….)
   b) PAR, NOR studies for the reaction conditions (Reagents Qty’s, Temperature range. Maintenance time, RPM,etc…)
   c) What if studies for longer hours of reaction maintenance (both temp and time), RPM (High & Low), etc…
   d) Blank reactions / studies data and conclusion.
   e) “If-condition” in-case of failure of the IPC / reaction incompletion.
   f) MIXIT simulation / observation / conclusion

2) Quenching / work-up:
   a) Basic observations (Qty of layers, Mass balance in Aq and Org layers, etc…)
   b) Impact of mixing on layer sepeation
   c) MIXIT simulation / observation / conclusion

3) Distillation:
   a) Basic observations (Solvent, Thermal degradation if any, etc….)
   b) Vapour pressure data (azeotrope data)
   c) Avoid dry distillation and generate data for impact on quality and yield if solvent volumes remains.
   d) What-if studies for longer hour of distillation (both temp and time) impact on quality and yield of the product.

4) Crystallisation:

a) Basic observations (Solvent used, Type of Crystallisation)

b) Solubility data generated for the solvent selected

c) What-if studies for longer heating time / cooling time, RPM (High & low), Type of Agitator, %Occupancy (High & low), etc….

d) MIXIT simulation / observation / conclusion

5) Filtration:

a) Basic observations (Solvent(s), wet weight, cake thickness, filter area / dia used, etc….)

b) Pocket filter studies (If settling time is >10-20 Sec) and conclusion for scale-up

c) Basis for scale-up of filter equipment (filtration area calculation, cake thickness, pH, etc….)

d) Physical properties like bulk density, morphology, etc….

6) Drying:

a) Basic observations (Solvent / water, temperature, Spec limits, etc….)

b) What-if studies for longer hours of drying (both temp and time) impact on quality of the product.

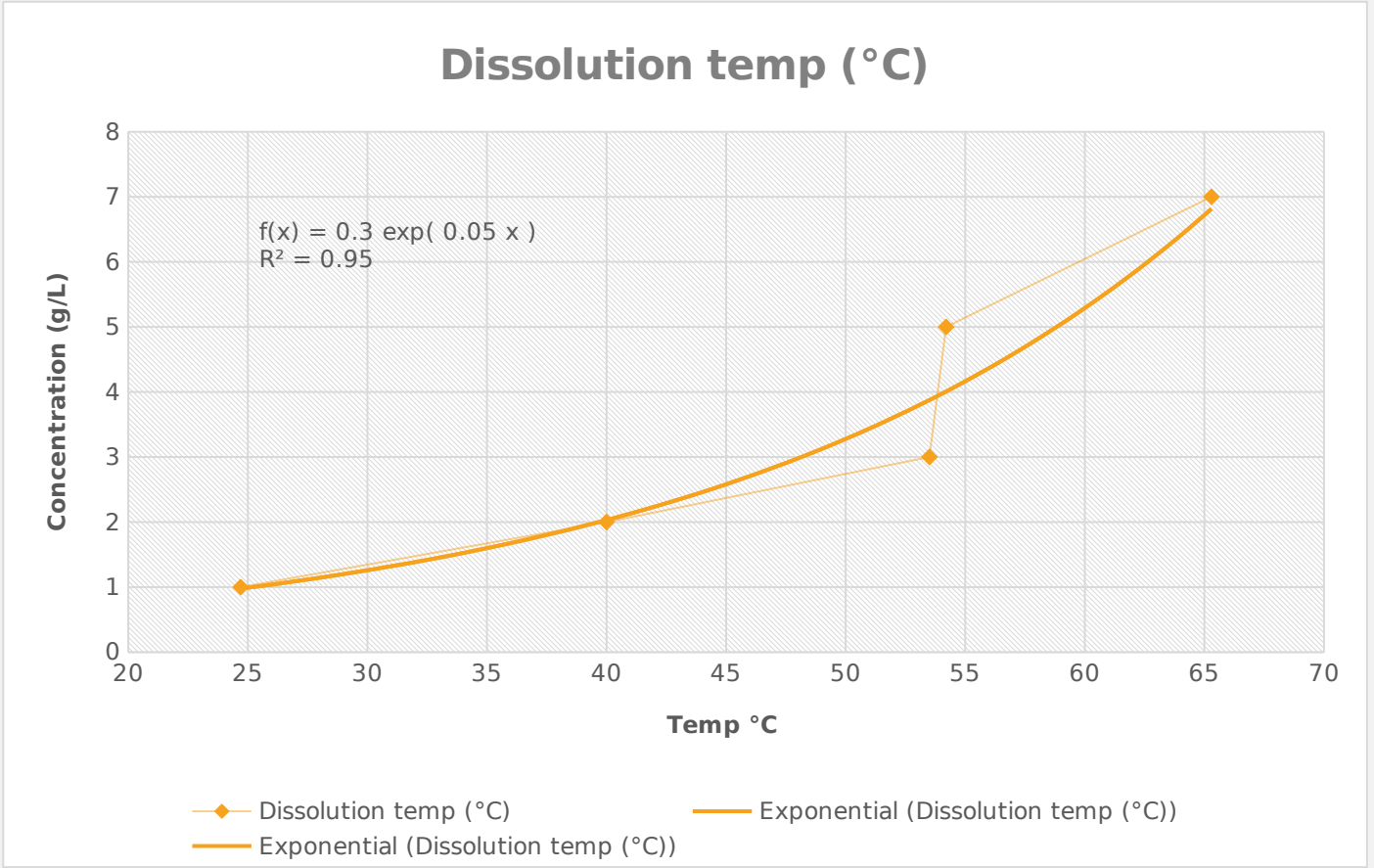
c) Physical properties of the wet and dry solid like bulk density, morphology, XRD, etc…

d) Tray loading and impact with increase scale.

e) Studies w,r,t type of drying like ATD, VTD and RCVD and Powder safety studies.

SOLUBILITY DATA - CRYSTALLISATION

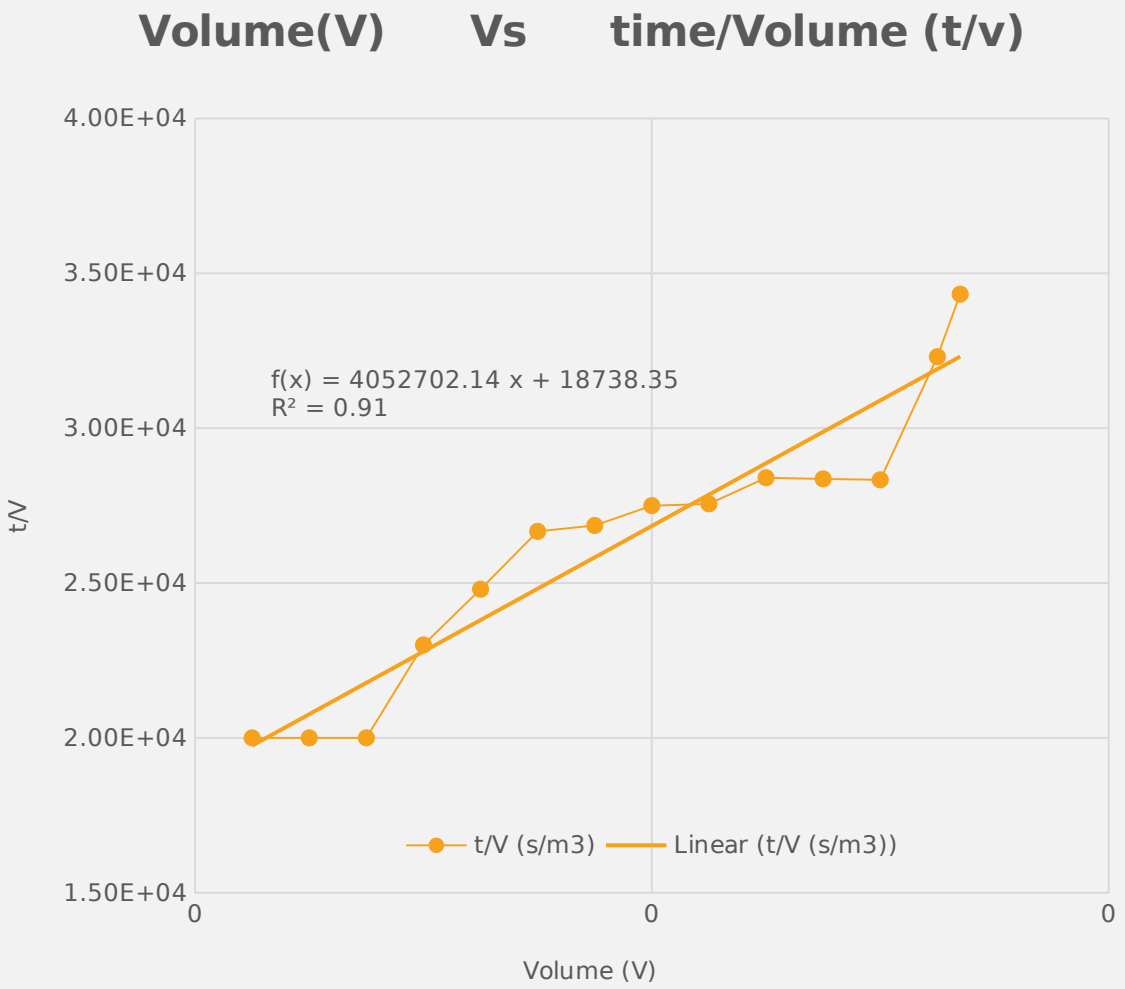
S.No	Concentration (g/L)	Dissolution temp (°C)	Nucleation temp (°C)	MSZW* °C
1	1	24.7	19.2	5.5
2	2	40	36.7	3.3
3	3	53.5	44.3	9.2
4	5	54.2	52.2	2
5	7	65.3	64.3	1



FILTRATION DATA – POCKET FILTER STUDIES

S.No	Parameter	Filtration Data	Unit
1	Batch size	0.3	kg
2	Diameter of Buckner funnel	0.19	m
3	Cake thickness	0.032	m
4	Filtrate volume	0.00335	m3
5	surface area of filter	0.0283385	m2
6	Time of filtration	1.92	min
7	Viscosity	1.00	m. Pas
		1.00E-03	kg/m.s
8	Vacuum	600	mmHg
		8.16E+03	kg/m2
9	Filter cloth size	5	micron
10	Concentration	89.55	kg/m3
11	Wet cake weight	0.454	kg
12	Bulk density	500.64	kg/m3
13	Filtration flux	**61.68**	Lpm/m2
14	Resistance of Filter cloth (Rm)	**4.3262E+09**	1/m
15	Specific Cake Resistance (α)	**5.92E+08**	m/kg

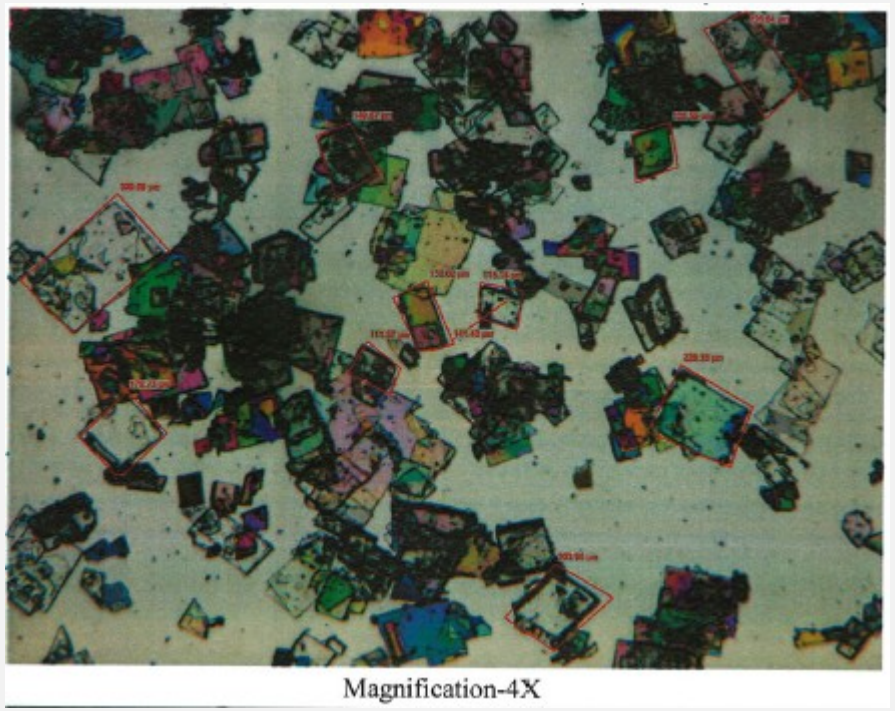
Time (s)	volume of filtrate (ml)	volume of filtrate (m3)	t/V (s/m3)
5	250.00	0.00025	2.00E+04
10	500.00	0.0005	2.00E+04
15	750.00	0.00075	2.00E+04
23	1000.00	0.001	2.30E+04
31	1250.00	0.00125	2.48E+04
40	1500.00	0.0015	2.67E+04



From Graph:

Intercept	1.87E+04	s/m3
Slope (t/V2)	4.05E+06	s/m6

FILTRATION DATA – MICROSCOPIC IMAGES

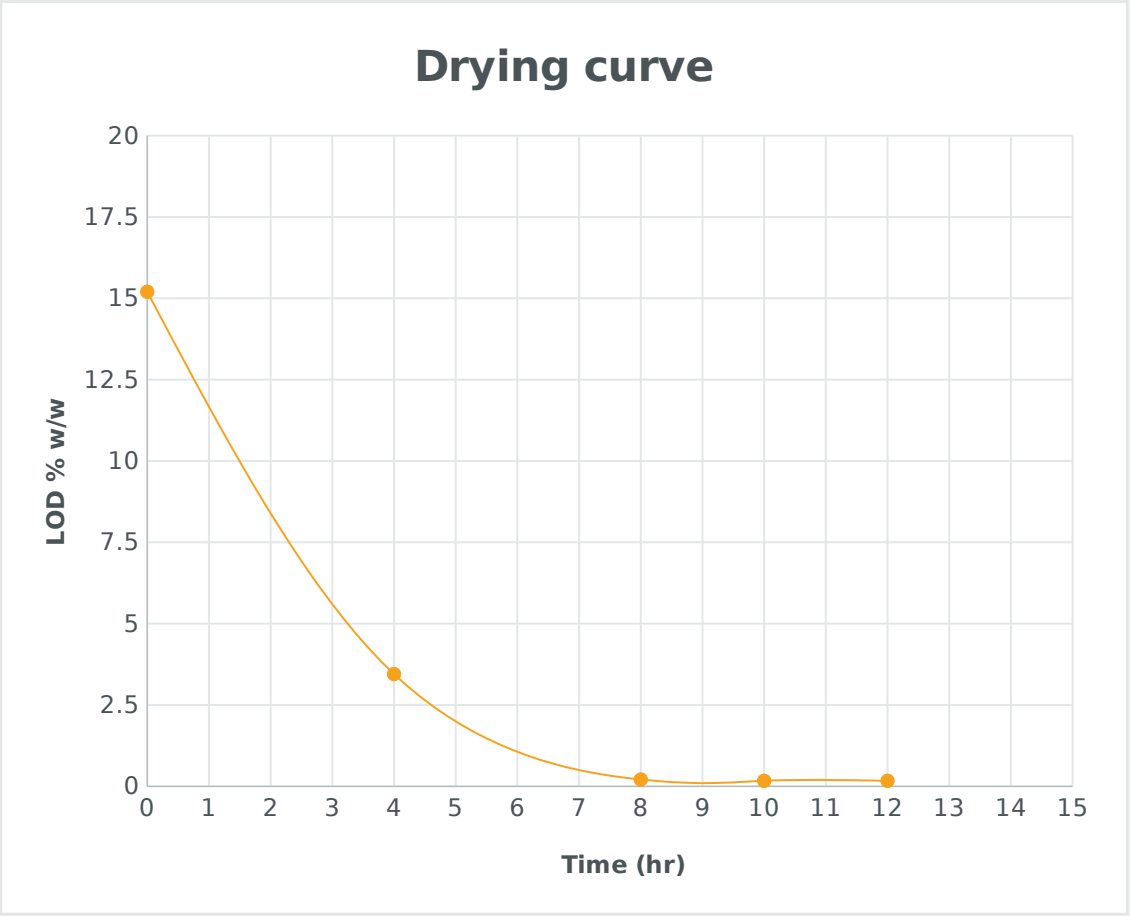


Magnification-4X

Present a comparison between lab & kilolab batches
Conclusion from the comparison

DRYING DATA

Vacuum Tray drier		
Description	**UOM**	**Batch 1**
Batch size	Kg	3.0
Utility inlet temperature	°C	57
Utility outlet temperature	°C	56.1
LOD of wet cake	% w/w	15.57
Vacuum	mm Hg	715
Wet cake depth in tray (if possible try to keep it nearly same at all the scales)	mm	~ 5 mm per tray (4 trays)
Mass of wet cake	Kg	3.96
LOD of dry powder (% w/w)	% w/w	0.17%
Drying cycle time (hrs)	hrs	12
Weight of Dry Powder	kg	3.35
Dry material Bulk Density	kg/m3	396



PROCESS SAFETY DATA

1) RC1e Experiment data / conclusions
2) DSC / RSD data for high temp reactions, distillation and drying powders

EQUIPMENT PROPOSAL FOR COMMERCIAL SCALE

Operation	Kilo lab equipment	Plant scale equipment	Justification

Thank you!